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# Indian Standard

# METHODS OF CHEMICAL ANALYSIS OF FERRO-MOLYBDENUM

## PART 7 DETERMINATION OF ALUMINIUM

भारतीय मानक

फेरो-मालिबडेनम के रासायनिक विश्लेषण की पद्धति

भाग 7 एलुमिनियम ज्ञात करना

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NEW DELHI 110002

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**Price Group 1**

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, SMDC 2

## FOREWORD

This Indian Standard ( Part 7 ) was adopted by the Bureau of Indian Standards on 17 October 1988, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferro-alloys' covers the chemical analysis of different ferro-alloys, such as, ferrosilicon, ferrochromium, ferro manganese and speigeleisen, silico manganese, ferromolybdenum ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten While reviewing this standard, the sectional committee had decided that a separate series for each ferroalloy be prepared. Accordingly IS 1559 in its various parts was published for the chemical analysis of ferrosilicon.

The chemical analysis of ferromolybdenum is covered into series of parts of this standard. With the publication of these parts, the analysis of relevant constituent prescribed for ferromolybdenum in IS 1559 : 1961 will be superseded. This part covers the determination of aluminium. The other parts are:

Part 1 Determination of molybdenum
Part 2 Determination of total carbon
Part 3 Determination of silicon
Part 4 Determination of sulphur
Part 5 Determination of phosphorus
Part 6 Determination of copper

In this revision, determination of aluminium by atomic absorption spectrophotometric method has been incorporated.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'.

*Indian Standard*

# METHODS OF CHEMICAL ANALYSIS OF FERRO-MOLYBDENUM

## PART 7 DETERMINATION OF ALUMINIUM

### 1 SCOPE

This standard ( Part 7 ) covers the atomic absorption spectrophotometric method for determination of aluminium in ferro-molybdenum.

### 2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

| *IS No.* | *Title* |
|---|---|
| IS 264 : 1976 | Specification for nitric acid |
| IS 1472 : 1977 | Method of sampling ferro-alloys for determination of chemical composition |

### 3 SAMPLING

Samples shall be drawn and prepared in accordance with IS 1472 : 1977.

### 4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and deionized water shall be employed in the test.

### 5 DETERMINATION OF ALUMINIUM IN FERRO-MOLYBDENUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRIC ( AAS METHOD )

**5.1 Outline of the Method**

Sample is dissolved in acids. Insoluble residue is hydrofluorized, fused with potassium pyrosulphate and taken up with the main solution. Sample solution is aspirated into nitrous oxide acetylene flame of AAS and aluminium is determined at 396·2 nm.

**5.2 Reagents**

**5.2.1** *Dilute Hydrochloric Acid,* 1 : 1 and 1 : 50 ( *v/v* ).

**5.2.2** *Concentrated Nitric Acid,* rd=1·42 ( conforming to IS 264 : 1976 )

**5.2.3** *Dilute Sulphuric Acid,* 1 : 1 ( *v/v* )

**5.2.4** *Hydrofluoric Acid,* 40 percent

**5.2.5** *Potassium Pyrosulphate,* solid

**5.2.6** *Potassium Pyrosulphate Solution*

Transfer 5 g of potassium pyrosulphate in a platinum crucible and fuse by ignition. Dissolve in water and dilute to 50 ml ( this solution shall be used within 24 hours ).

**5.2.7** *Iron Solution* ( *20 mg Fe/ml* )

Weigh 2·00 g of pure iron ( 99·99 percent pure ) in a 250 ml beaker. Add 10 ml of dilute hydrochloric acid ( 1 : 1 ) and heat. Cool, add 1 ml of hydrogen peroxide dropwise and 1 ml of water and evaporate to dryness. Add 2 ml of dilute hydrochloric acid ( 1 : 1 ) and cool. Transfer the contents to separating funnel ( 100 ml ). Add the same quantity of methyl isobutyl ketone ( MIBK ) as the solution in the separating funnel and shake for 1 min. Let the layers to separate and discard the aqueous layer. Add 2 ml of dilute hydrochloric acid ( 1 : 1 ) to the organic phase and shake again for 30 seconds and separate into two layers. Discard again the aqueous layers. Add 10 ml of water and shake vigorously for 30 s and let the phases to separate. Transfer the water layer to the original beaker. Repeat operation again with the organic phase and combine the water extract in the above beaker. Heat the solution for boiling and add 1 ml nitric acid. Cool and add 10 ml of dilute hydrochloric acid ( 1 : 1 ), and transfer to 100 ml volumetric flask and make up.

**5.2.8** *Molybdenum Solution* ( 25 mg Mo/ml )

Dissolve 23 g of ammonium molybdate $[ ( NH_4 )_6Mo_7O_{24} . 4H_2O ]$ in 180 ml of water. Transfer to 500 ml volumetric flask and make up.

**5.2.9** *Standard Aluminium Solution* ( 1 ml = 0·1 mg Al )

Transfer 1·000 g of aluminium ( purity 99·95 percent, *Min* ) into an Erlenmeyer flask. Add 50 ml dilute hydrochloric acid ( 1 : 1 ) and put a funnel on the mouth of the flask. After reaction has subsided, add 1 ml of hydrogen peroxide and boil. Cool and transfer to 1 litre volumetric flask. Add 50 ml of concentrated hydrochloric acid and make up.

Transfer 10 ml of the solution into 100 ml volumetric flask and make up.

### 5.3 Procedure

### 5.3.1 *Preparation of Sample Solution*

Weigh 0·500 0 g of sample in a 250 ml beaker and cover with a watch glass. Add 20 ml of dilute hydrochloric acid ( 1 : 1 ), 5 ml of concentrated nitric acid and cover with a watch glass and heat gently to complete the dissolution. Add 10 ml of dilute sulphuric acid ( 1 : 1 ), and heat until white fumes of sulphur trioxide are evolved. Cool and add 20 ml of dilute hydrochloric acid ( 1 : 1 ) again. Dilute to 50 ml with warm water and filter through a Whatman filter paper ( 541 ). Wash the residue 4 to 5 times with warm dilute hydrochloric acid ( 1 : 50 ) until iron disappears in the washings and wash again with warm water till acid free. Collect the filtrate and washings into a 200 ml beaker ( *A* ) and evaporate to 20 ml. Transfer the residue along with filter paper to a platinum crucible and incinerate the filter paper by gradually heating it. Cool, moisten the residue with 2 to 3 drops of dilute sulphuric acid ( 1 : 1 ) and add 5 ml of hydrofluoric acid and cautiously evaporate to dryness. Add 1·0 g of potassium pyrosulphate to the platinum crucible and cover it with a cap, gradually raise the temperature and completely fuse the residue at 700°C. Cool, dissolve the fused residue on the reserve filtrate ( beaker *A* ) and wash the crucible. Cool the solution and transfer to 50 ml volumetric flask and make up ( Test solution ).

### 5.3.2 *Calibration Solutions*

Take six 200 ml beakers. To each of these add 10 ml of iron solution ( **5.2.7** ) and 10 ml of molybdenum solution ( **5.2.8** ). Add 0, 5, 10, 15, 20 and 25 ml of standard aluminium solution ( **5.2.9** ) successively. Then add 10 ml each of concentrated nitric acid and dilute sulphuric acid ( 1 : 1 ) to each beaker. Evaporate the solutions to dense white fumes. Cool, add 20 ml of dilute hydrochloric acid ( 1 : 1 ) and 10 ml of potassium pyrosulphate solution ( **5.2.6** ). Cool, transfer to 50 ml volumetric flasks and make up.

### 5.3.3 *Atomic Absorption Measurements*

Adjust the atomic absorption instrument using instrument parameters recommended by the manufacturer. Light the burner, set the wave length 396·2 nm for determination of aluminium. Aspirate water until thermal equilibrium is established. Aspirate the calibration solutions and the test solution into nitrous oxide-acetylene flame of AAS in the order of increasing absorption starting with zero calibration solution. When a stable response has been obtained for each solution, record the readings. Aspirate water between each calibration solution and test solution. Repeat the measurement twice. Take the average values of the readings. Obtain the net absorbance of each calibration solution and test solution by substracting the average absorbance of zero calibration solution. Prepare calibration curve by plotting the net absorbance values of calibration solutions against mg of aluminium/ml. Convert the net absorbance value of test solution to mg of aluminium by means of the calibration curve.

### 5.3.4 *Calculation*

$$\text{Aluminium, percent} = \frac{A}{B} \times 100$$

where

$A$ = mass in g of aluminium in sample solution, and

$B$ = mass in g of sample taken for the test.

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